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ORIGINAL PAPER

Electrogeneration of hydrogen peroxide in seawater and application to disinfection

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Abstract The cathodic electrogeneration of hydrogen peroxide in seawater by means of oxygen reduction on a gas diffusion cathode was studied. The effects on the reaction yield of several operative parameters such as cell design, medium composition, anolyte concentration, pH and working potential were investigated. Results indicate that in a two-compartment cell notable concentrations of hydrogen peroxide are obtained with a constant yield in a wide range of charge. Lower catholyte pH values, obtainable by means of the anolyte choice, mitigate the decrease in the efficiency due to cathode fouling. Application of hydrogen peroxide electrogeneration to seawater disinfection was also tested. Comparative tests conducted using both commercial and electrogenerated hydrogen peroxide, either alone or combined with iron in Fenton's treatment, are also presented.

Keywords Disinfection · Gas diffusion electrode · Hydrogen peroxide · Oxygen reduction · Seawater

1 Introduction

Water disinfection can occur using a range of different treatments, including physical [1], thermal [2] or chemical methods. Currently the most widely used methods are chlorination [3, 4] and ozonation [5, 6]. Despite the effectiveness of these two treatments, chlorination has the disadvantage of generating hazardous and carcinogenic by-products [7–9], and ozonation has a high operating cost [10]. Alternative technologies have been developed to meet water quality objectives while maintaining safety and controlling costs. These include ultrafiltration [11], disinfection by copper and silver ions [12], cavitation [13] and photocatalytic processes [14]. Electrochemical technologies have been recently considered as a promising alternative to deal with several topics of environmental concern in water and wastewater treatment [15]. Electrochemical disinfection has proven to be effective in inactivating different microorganisms by the formation of highly reactive species [16–19], and has the advantage of eliminating chemical additions to the water.

Among all the oxidant agents that can be electrogenerated through an electrochemical process, hydrogen peroxide is one of the most environmentally friendly. It decomposes into oxygen and water and does not release any harmful compounds after reaction [20]: thus, it has a low residual impact. Although many different chemical processes are known for producing hydrogen peroxide, in situ electrogeneration has the significant advantage of avoiding storage, transportation and handling of highly concentrated solutions. This process occurs on graphite cathodes through the reduction of molecular oxygen [21–23]. The overall process, which also involves the formation of numerous radical species such as superoxide radicals $O_2^{\bullet -}$ and hydroperoxide radicals $HOO^{\bullet}$, can be described by reaction 1:

$$O_2 + 2H^+ + 2e^- \rightarrow H_2O_2 \qquad (1)$$

Due to the development of innovative materials, the in situ electrogeneration of hydrogen peroxide, using the cathodic reduction of molecular oxygen, is a competitive alternative to traditional production methods. In particular, the use of either a catalysed [24] or an uncatalysed [25, 26]

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gas diffusion electrode, instead of the traditional graphite cathode, has resulted in increased production efficiency and decreased cost [27].

While several studies are available in the literature regarding the disinfection of drinking water, less information can be found about the application to seawater for the treatment of the so-called "ballast water" [28, 29]. Ballast water is brought on board a vessel as a replacement for cargo to stabilize a ship. It has been recognized as a major vector for the unintentional transfer of exotic aquatic organisms between and within many parts of the world: thus, it is an increasing threat to the biodiversity of the sea and particularly of coastal areas. Additional applications are: disinfection treatment in aquaculture and in seawater swimming pools.

In the present work, the electrogeneration of hydrogen peroxide in seawater was studied by investigating the main operative parameters, such as cell design, medium composition, working potential, anolyte concentration and pH.

The application to disinfection was then evaluated by monitoring Escherichia coli and total bacterial charge as indicators of biological contamination. A comparison among several treatments was performed by investigating the efficiency of both the electrogenerated and commercial hydrogen peroxide, and by evaluating the improvement that results from combining hydrogen peroxide with ferrous ions in Fenton's reaction (Eq. 2), either chemically or electrochemically generated [30]. This procedure is one of the most powerful oxidation methods that can be successfully applied to the treatment of water and wastewater [31, 32]

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\bullet} + OH^- \qquad (2)$$

2 Experimental

2.1 Hydrogen peroxide production

A potentiostat (AMEL 2051) was employed for controlled electrolyses carried out in a batch Plexiglas electrolyser where a cation exchange membrane (Nafion® 324) was used to separate cathodic and anodic compartments. The cathode, of surface area about 5 cm^2, was a carbon "Vulcan" gas diffusion electrode supplied by De Nora Tecnologie Elettrochimiche, fed with pure O_2; a platinum wire and a saturated calomel electrode (SCE) were respectively used as anode and reference electrode. The apparatus was described previously [27]. Where not differently indicated, electrolysis was conducted at the controlled potential of −0.9 V vs SCE, the catholyte consisted of 100 ml of synthetic seawater prepared from a certified mix of salt, and the anolyte was a 100 ml solution of sodium perchlorate 0.01 M. No supporting electrolyte was added to the catholyte, since conductivity was guaranteed by the salinity of seawater. The solution was agitated at 1250 rpm by a magnetic stirrer and was maintained at 25 °C using a recirculating water bath.

The current efficiency, defined as the ratio of the hydrogen peroxide produced to that theoretically obtained by means of Faraday's law, was calculated according Eq. 3:

$$CE(\%) = \frac{[H_2O_2]_{exp}}{[H_2O_2]_{theor}} \times 100 = \frac{nF[H_2O_2]_{exp}}{Q \times MW \times 10^4} \times 100 \qquad (3)$$

where

n = number of equivalents
F = Faraday constant (96485 C mol^{-1})
$[H_2O_2]_{exp}$ = experimental concentration (mg L^{-1}) at a given time t
Q = charge (coulomb) at a given time t
MW = hydrogen peroxide molecular weight
10^4 = conversion factor

All the experiments were conducted at least in triplicate.

2.2 Disinfection

These tests were carried out on synthetic seawater enriched with a standardised content of bacterial species from an activated sludge pilot plant. Removal was calculated as the percentage ratio between the variation in the total bacterial charge and the initial total bacterial charge (Eq. 4).

$$R = \frac{C_0 - C_t}{C_0} \times 100 \qquad (4)$$

where C_0 is the initial bacterial charge and C_t is the bacterial charge at the considered time.

All the experiments were conducted at least in triplicate.

2.2.1 Electrochemical treatment procedure

The electrolyses were performed under the same operating conditions used for the hydrogen peroxide production tests. When required, in Fenton's treatment, 25 mg L^{-1} of iron was added (as heptahydrate iron sulphate $FeSO_4 \cdot 7H_2O$) at the start of the electrolysis.

2.2.2 Chemical treatment procedure

Oxidation tests were performed at ambient temperature and under conditions of constant agitation on 100 ml solution after adjustment to pH 2.8 by means of hydrochloridric acid. Experiments were carried out by dosing 300 mg L^{-1} of commercial hydrogen peroxide, and, in Fenton's treatment, adding further 150 or 100 mg L^{-1} of iron (as heptahydrate iron sulphate $FeSO_4 \cdot 7H_2O$).

2.3 Analysis

The pH was monitored by using a continuous Crison 421 pH-meter. Hydrogen peroxide concentrations were determined reflectometrically by means of Merck analytical tests. Biological analysis consisted in the count of the total bacteria and Escherichia Coli charge, implemented through filtration and growing in sealed sterile monitors (Millipore). Bacterial cultures were counted after incubation (Millipore) at 32 °C for 24 h.

2.4 Materials

Reagents were supplied by the Carlo Erba Company and used in their commercially available form with no further purification. As preliminary experiments indicated no difference in behaviour between synthetic and real seawater, a synthetic solution, prepared by dissolving certificated salts in distilled water, was used to enhance the reproducibility of the trials.

3 Results and discussion

3.1 Hydrogen peroxide production

3.1.1 Test in undivided cell

To evaluate the possibility of producing hydrogen peroxide in an undivided cell a series of tests were preliminarily conducted on seawater, both with and without separation between the two electrolytic compartments realised by means of a cationic membrane. The data reported in Table 1, as already highlighted [33], showed that the current efficiency and the hydrogen peroxide production in the divided cell were notably higher than those in the undivided cell, where evidently the simultaneous anodic decomposition of the produced hydrogen peroxide occurred. In particular, in the divided cell, except for an initial lower value, an almost constant current efficiency near 70% was observed, thus indicating that the process, in the investigated range of charge, was under current control.

Table 1 Hydrogen peroxide production and current efficiency as a function of the charge in undivided and divided cell

Undivided cell			Divided cell		
Charge (C)	[H_2O_2] (mg L^{-1})	CE (%)	Charge (C)	[H_2O_2] (mg L^{-1})	CE (%)
0	0	–	0	0	–
108	22	11.6	163	162	56.4
226	42	10.5	518	624	68.4
466	50	6.1	693	835	68.4
687	78	6.4	1,055	1,220	65.6
			1,387	1,550	63.4
			1,700	2,190	73.1
			1,912	2,365	70.2
			2,400	2,900	68.6

Catholyte: seawater; Anolyte: $NaClO_4$; 0.01 M; Potential, −0.9 V vs SCE

The lack of separation between the two electrode compartments not only prevented hydrogen peroxide from accumulating but also led to the formation of high concentrations of undesired compounds, mainly chlorine and hypochlorite.

According to these results, a divided scheme was then adopted for this study, although the use of a membrane involved a more difficult reactor design, additional costs and higher cell resistance.

3.1.2 Effect of chloride concentration

Marine water is characterised by a high concentration of chloride ions (Table 2), whose effect of quencher versus radicals is well known [34]. Their presence could negatively affect the hydrogen peroxide electrogeneration, whose mechanism involves the participation of several radical species.

On the other hand, chlorides may inhibit the H_2 evolution (Eq. 5) thus promoting H_2O_2 generation as also confirmed by recent studies [35, 36].

$$2H^+ + 2e^- \rightarrow H_2 \qquad (5)$$

A series of tests were then conducted to study the influence of the concentration of this species on the hydrogen peroxide production, in a range of NaCl concentration varying between 3.5 and 35 g L^{-1}, respectively corresponding to the minimum useful concentration to reach a sustainable current intensity and the average value of marine salinity. The results were then compared with a test conducted in seawater. Figure 1 showed that the chloride concentration did not negatively affect the production yield of hydrogen peroxide. In contrast, a higher concentration of chloride

Table 2 Seawater average composition

Salt	Concentration (g L^{-1})
NaCl	22
$MgCl_2$ 6 H_2O	9.7
Na_2SO_4	3.7
$CaCl_2$	1.0
KCl	0.65
$NaHCO_3$	0.20
H_3BO_3	0.023

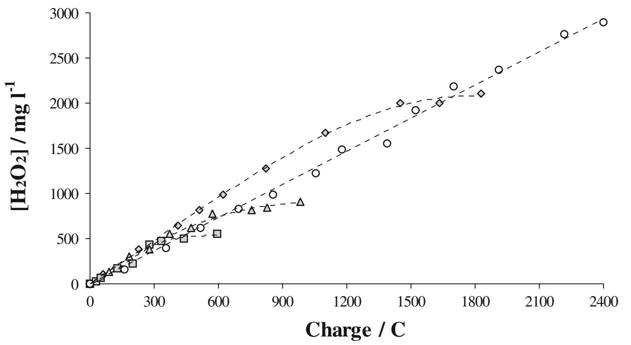

Fig. 1 Hydrogen peroxide production versus charge as a function of the NaCl concentration: (■) 3.5 g L^{-1}, (▲) 15.0 g L^{-1}, (◆) 35.0 g L^{-1}, (○) seawater. Anolyte: $NaClO_4$ 0.01 M, potential: −0.9 V vs SCE

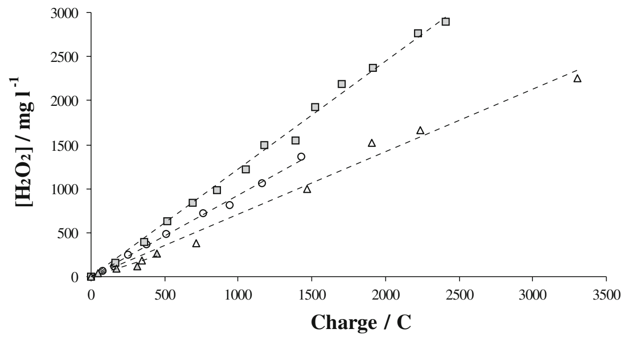

Fig. 2 Hydrogen peroxide production versus charge as a function of the working potential: (▲) −0.5 V vs SCE, (○) −0.7 V vs SCE, (■) −0.9 V vs SCE, (△) −1.1 V vs SCE. Catholyte: seawater, anolyte: $NaClO_4$ 0.01 M

postponed the asymptotic trend caused by the predominance of side reactions (mainly Eq. 5). In addition, because of the different ionic strength of those solutions (leading to average values of conductivity of 5.5 mS, 22 mS and 45 mS respectively for the 3.5, 15 and 35 g L^{-1} of NaCl solution), a higher concentration corresponded with higher current intensity and subsequently a higher production rate. In seawater, despite a linear trend in a wide range of charge, slightly lower yields were found.

3.1.3 *Effect of solution composition*

During electrolysis in seawater deposition of a thick layer of white salts covering the cathode surface occurred. Analysis of some scales, sampled from the cathode cloth and carried out by ionic chromatography and SEM-EDX microscopy, indicated that this deposit was composed mainly of magnesium hydroxides. Traces of sodium and calcium ions were also detected. The formation of this insoluble hydroxide, according to Eq. 6, was due to the reaction between the magnesium, which is present as chloride in a consistent concentration in marine water (see Table 2), with OH$^-$ ions, which are available in large amounts in the proximity of the cathode.

$$Mg^{2+} + 2OH^- \rightarrow Mg(OH)_2 \downarrow \tag{6}$$

3.1.4 *Effect of working potential*

To investigate the influence of cathode potential on hydrogen peroxide production, a series of tests was conducted in the range −0.5 and −1.1 V vs SCE. In Fig. 2, hydrogen peroxide production is shown as a function of charge. Since all the electrolyses were stopped after 450 minutes, the length of each curve was representative of the reaction rate and therefore of the current density at which reaction occurred. The best performance was obtained for −0.9 V vs SCE, while when the cathodic potential increased further the production efficiency dropped notably due to the occurrence of side reactions, in particular hydrogen evolution (Eq. 5) and oxygen reduction to water (Eq. 7).

$$O_2 + 4H^+ + 2e^- \rightarrow 2H_2O \tag{7}$$

Both these reactions induced a local pH increase near the electrode surface favouring the precipitation of magnesium hydroxide and thus leading to the formation of a thicker layer of salts. In these working conditions the cathode presented a faster deterioration which limited its lifetime to a few tens of hours.

Electrolysis conducted at −0.5 V vs SCE gave low efficiency, together with an unacceptable reaction rate. A potential of −0.9 V vs SCE was adopted for all the following tests.

3.1.5 *Effect of anolyte concentration*

In a divided cell the choice of the anolyte is an important parameter. In this study sodium perchlorate was used so that no reaction other than water electrolysis occurred (Eq. 8), chlorine being at its maximum oxidation state.

$$H_2O \rightarrow {}^1\!/_2 O_2 + 2H^+ + 2e^- \tag{8}$$

The influence of anolyte concentration on hydrogen peroxide production in seawater was investigated in the range 0.01–1 M. As indicated in Fig. 3 better efficiencies were found when the anolyte was less concentrated.

A study of the pH trend of the catholyte during these runs, as shown in Fig. 4, highlighted a very different behaviour depending on the $NaClO_4$ concentration. In particular, when an anolyte concentration of 0.01 M was used, the catholyte pH quickly dropped from the initial value of 8–9 to very acidic values, while a slight increase was observed when its concentration was 1 M. In the first case the whole electrolysis occurred at a pH of 2; in the

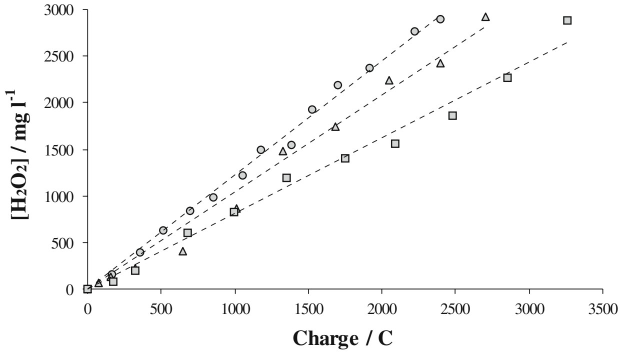

Fig. 3 Hydrogen peroxide production versus charge as a function of the $NaClO_4$ concentration: (●) 0.01 M, (▲) 0.1 M, (■) 1 M. Catholyte: seawater, potential: −0.9 V vs SCE

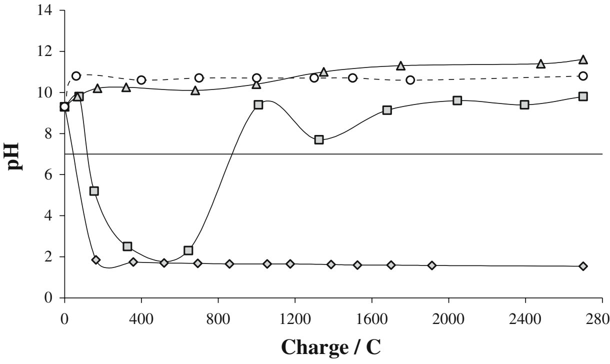

Fig. 4 pH trend versus charge as a function of the $NaClO_4$ concentration: (◆) 0.01 M, (■) 0.1 M, (▲) 1 M, (○) NaCl 35.0 g L^{-1}. Catholyte: seawater, potential: −0.9 V vs SCE

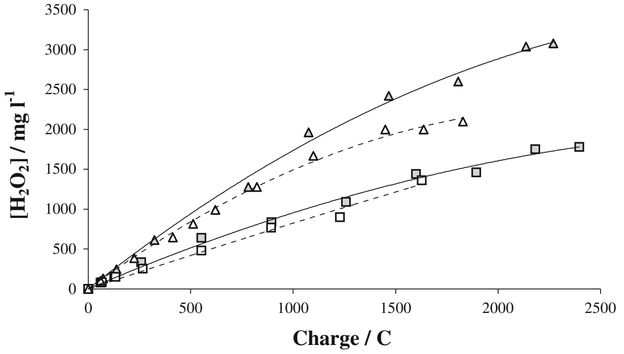

Fig. 5 Hydrogen peroxide production versus charge as a function of the pH. In sulphate solutions: (▲) pH 12, (■) pH 1, and in chloride solutions: (△) pH 10, (□) pH 1. Anolyte: $NaClO_4$ 0.01 M, potential: −0.9 V vs SCE

second case at a pH of 11. The anolyte intermediate concentration led to an alternate trend which reached a minimum after two hours (corresponding to the passage of about 500 C), then it grew rapidly to alkaline values which were then maintained until the end of the reaction. Comparison with pH data obtained at the same operating conditions in 35 g L^{-1} NaCl, highlighted a quite different behaviour since, in this case, independently of the anolyte concentration, the catholyte always turned alkaline (for clarity, only one curve is shown in the figure).

3.1.6 *Effect of pH*

Comparing the data reported in Figs. 3 and 4 hydrogen peroxide production in seawater is favoured at acidic pH. Since contradictory data were available in the literature [23, 37] the influence of pH on hydrogen peroxide production was investigated in different media. A series of tests were then conducted respectively in chloride solutions and in sulphate solutions, characterised by a value of conductivity equal to that of seawater, that is 45 mS. In the first case the pH values investigated were 1 (obtained using HCl), 7 (using NaCl), and 10 (using a solution of NaCl corrected with NaOH). In the second case the pH values investigated were 1 (obtained using H_2SO_4), 7 (using Na_2SO_4), and 12 (using a solution of Na_2SO_4 corrected with NaOH).

The results confirmed the direct dependence of hydrogen peroxide production on pH, highlighting how the anomaly was to be found exclusively in seawater. For clarity, in Fig. 5 only curves observed at alkaline and acidic pH are shown given that the curves obtained at a neutral pH confirmed the general trend assuming an intermediate position.

3.1.7 *Discussion*

To better understand the overall process, three main reactions which occurred simultaneously in the bulk have to be considered. Firstly, hydrogen peroxide production in the cathodic compartment, Eq. 1, which subtracts H^+ ions contributing to the increase in the pH solution. Secondly, oxygen evolution in the anodic compartment, Eq. 8, which generates H^+ ions whose transfer towards the catholyte through the cationic membrane contributes to the catholyte acidification. Thirdly, when seawater is concerned, $Mg(OH)_2$ production, Eq. 6, which subtracts OH^- thus contributing to a catholyte pH decrease.

In the tests conducted on NaCl solutions, only Eqs. 1 and 8 take place, and therefore the fact that an alkaline pH was always found, clearly indicates that the acidification coming from the anolyte (Eq. 8) was insufficient to replace the consumption due to oxygen reduction (Eq. 1).

In the tests conducted in seawater, on the other hand, the contribution of reaction 5 involving $Mg(OH)_2$ production, seems to represent the crucial step in determining the pH conditions. This conclusion was confirmed by an additional electrolysis, not reported here, where the anolyte was $NaClO_4$ 0.01 M and the catholyte was a chloride solution of 35 g L^{-1}. As reported above, in these conditions, the catholyte pH quickly turned to alkaline values. Whereas, after an addition to the catholyte of an amount of $MgCl_2$ corresponding to that of seawater, its pH dropped to a value of 2.5 in less then 15 min. Only the simultaneous occurrence of Eq. 6 and the transfer of H^+ from the anolyte can cover the deficit of H^+ ions caused by Eq. 1, leading the catholyte to acidic pH. However, this effect actually occurs only for the lowest anolyte concentration. In fact, with increasing $NaClO_4$ concentration, excess Na^+ causes competition to transfer towards the catholyte between this species and hydrogen ions. For this reason, the contribution of the passage of the H^+ electrogenerated at the anode, despite the acidification coming from the $Mg(OH)_2$ precipitation, can be insufficient to counterbalance the alkalinity developed by hydrogen production thus leading to alkaline pH.

Thus, it is now possible to explain the influence of pH on the yield of hydrogen peroxide in seawater. In this medium, as already said, consistent deposition of magnesium hydroxides occurs, and appears particularly compact in alkaline conditions, when turbidity is also observed in the catholyte. This incrustation, causing electrode fouling, worsens the cathode efficiency. Higher acidity favours its solubilization contributing to notably better yields.

The following tests were then conducted with a concentration of perchlorate of 0.01 M, which allowed greater hydrogen peroxide concentrations to be reached with a saving of reagent.

3.2 Application to disinfection

Once the optimisation of the electrochemical hydrogen peroxide production in seawater was realised, the applicability of this process to disinfection was investigated.

In particular, the efficiency of a disinfection treatment by means of electrogenerated hydrogen peroxide was compared to a treatment using commercial H_2O_2 and a treatment where the effectiveness of this reagent was enhanced by the combined use of ferrous ions to realise the well known Fenton reaction. In Fig. 6, where the removal efficiency of both commercial and electrogenerated hydrogen peroxide are reported as a function of the time, the data indicated that the in situ electrogeneration of the reagent species results in better efficiency. In particular, 50% removal was obtained after half an hour, corresponding to a charge of about 100 C, when only 90 mg L^{-1} of hydrogen peroxide was found in the solution. Complete disinfection ocurred after 200 min, corresponding to a charge of about 500 C. At that time a residual hydrogen peroxide concentration of about 500 mg L^{-1} was found.

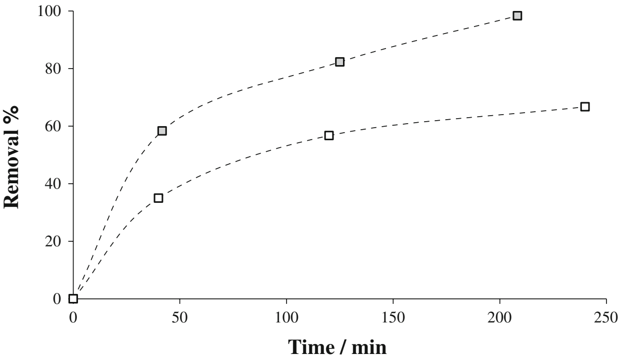

Fig. 6 Bacterial removal as a function of the time in seawater enriched with bacterial species: (■) electrogenerated H_2O_2, (□) commercial H_2O_2. Anolyte: $NaClO_4$ 0.01 M, potential: −0.9 V vs SCE

In the same operating conditions, treatment using commercial H_2O_2, although starting with a consistent H_2O_2 concentration (300 mg L^{-1}), showed a removal of about 50% after a two-hour contact time reaching, at most, a removal lower than 70% and only after four hours. After this time, in spite of a residual concentration of 250 mg L^{-1} of hydrogen peroxide, no further improvement was observed. The better efficiency of the electrogenerated hydrogen peroxide, even at a much lower concentration, is explained by the simultaneous action of different radical species involved in the production mechanism.

A further series of tests was then performed to investigate possible enhancement of the disinfection yield obtained by using hydrogen peroxide alone compared with that observed after the addition of ferrous ions in both chemical and electrochemical Fenton reactions. In those runs, a preliminary adjustment of the pH was required since Fenton's reaction occurs only in acidic conditions. Figure 7 shows that, with respect to electrogenerated hydrogen peroxide alone, addition of iron gives an increase in reaction rate without improving the total removal yield. In particular, in the chemical fenton treatment a removal higher than 80% was reached after a few minutes but no further improvement was noted after that time, not even by doubling the iron dosage, thus preventing the

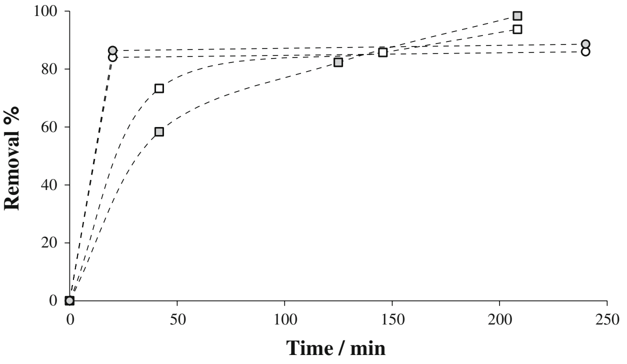

Fig. 7 Bacterial removal as a function of the time in seawater enriched with bacterial species: (■) electrogenerated H_2O_2, (□) electro-Fenton, (○) chemical Fenton $[H_2O_2] = 300$ mg L^{-1}, $[Fe^{2+}] = 25$ mg L^{-1}, (●) chemical Fenton $[H_2O_2] = 300$ mg L^{-1}, $[Fe^{2+}] = 50$ mg L^{-1}. Anolyte: $NaClO_4$ 0.01 M, potential: −0.9 V vs SCE

solution from complete disinfection. This behaviour is explained by considering that, in the chemical treatment the reaction occurred instantaneously with no possibility of regenerating the reagents. In an electro-Fenton treatment, continuous in-situ production of H_2O_2 and simultaneous iron regeneration notably increases the removal efficiency. However, despite an initially higher yield, after the first two hours the electro-Fenton treatment showed a trend quite similar, even if not better, to that performed by means of electrogenerated H_2O_2, with the additional disadvantage of involving the dosage of chemicals and an initial acidification step.

4 Conclusions

The possibility of producing hydrogen peroxide in seawater and the application to disinfection was proven. In a divided cell the production efficiency was not negatively affected by chloride ions while the main problem was the deposition of a $Mg(OH)_2$ layer on the electrode surface which caused a decrease in the production yield and hastened the cloth deterioration. The precipitation of this hydroxide could be prevented by working at a cathodic potential of -0.9 V vs SCE, which allowed the best yield to be obtained at a higher rate. The $Mg(OH)_2$ precipitation was also limited by working at acidic pH which can be achieved without the addition of chemicals taking advantage of the anolyte concentration.

Application of this process to seawater disinfection is promising considering that complete removal of the total bacterial charge was achieved without requiring chemical dosage and without leaving residual species after treatment.

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